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Indian Standard

TEXTILE DYESTUFFS — DETERMINATION OF SOLUTION STABILITY OF REACTIVE DYES IN PRESENCE OF ELECTROLYTES

ICS 59.040

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

September 1997 **Price Group 2**

Dyestuffs Sectional Committee, TX 17

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Dyestuffs Sectional Committee had been approved by the Textile Division Council.

Reactive dyes are commonly used for dyeing and printing of cellulosic materials by textile industry in the presence of electrolytes in batchwise and continuous dyeing process. Solution stability of reative dyes in the presence of electrolytes is an important characteristic to ensure proper dyeing. This standard is based on ISO 105 - 208 : 1995 'Textiles — Tests for colour fastness — Part 208 : Determination of solubility and solution stability of reactive dyes in the presence of electrolytes'

There are number of factors affecting the results and are given in Annex A for information.

Indian Standard

TEXTILE DYESTUFFS — DETERMINATION OF SOLUTION STABILITY OF REACTIVE DYES IN PRESENCE OF ELECTROLYTES

1 SCOPE

This standard prescribes a method for the determination of the solution stability of reactive dyes for use in batchwise and continuous dyeing process in the presence of electrolytes.

2 SAFETY PRECAUTIONS

2.1 It is the user's responsibility to use safe and proper techniques in handling materials and equipment. Manufacturers must be consulted for specific details such as safety data sheets and other recommendations.

2.2 Good laboratory practices have to be followed. Working with vacuum and chemicals (including dyes) requires safety measures such as wearing safety glasses, goggles or face shields, gloves, aprons and dust control.

3 PRINCIPLE

Several solutions of known concentration, including the solution stability limit, of the dye to be tested are prepared at a specified temperature in accordance with the dye manufacturer's recommendation. The dye solution is adjusted to the desired test temperature. A specified amount of the desired electrolyte solution is added and the resultant solution either filtered immediately or stored for a specified time at a specified temperature and subsequently filtered under suction.

The solubility and/or solution stability limits of the dye solution to the addition of electrolyte are determined by visual assessment of the filter residues and the measured flow-through time of the filtrate.

4 APPARATUS AND REAGENTS

4.1 Wide-mouthed Erlenmeyer flask, capacity 500 ml.

4.2 Thermostatically controlled heating bath, with magnetic stirring bar 40 mm long by 6 mm diameter, speed of stirrer 500 rev/min to 600 rev/min.

4.3 Water bath, with temperature regulator (heating/cooling) for adjusting the storage temperature.

4.4 Thermostatic device with circulation pump to adjust temperature of Nutsch filter (Buchner funnel).

4.5 Heatable double-walled Nutsch filter (Buchner funnel) of stainless steel or porcelain of inner diameter 70 mm, capacity at least 200 ml, having more than 100 holes with a total surface area of holes (evenly distributed) of not less than 200 mm^2.

4.6 Vacuum Apparatus

4.6.1 Suction bottle, capacity 1 litre to 2 litres.

4.6.2 Piston or membrane pump of sufficiently high suction capacity to create a full vacuum of at least 50 kPa under pressure.

4.6.3 Apparatus to adjust and maintain a given vacuum, preferably coupled with a manometer.

4.7 Stop-watch, to measure flow-through time.

4.8 Circular filter paper, 70 mm ± 2 mm diameter (Whatman No. 2).

4.9 Electrolyte solution (stock solution) prepared using distilled water or fully demineralized water according to the concentrations given in Table 1 or such that the required quantity of desired electrolyte is contained in 50 ml of solution.

NOTES

1 The reproducibility of the test method depends on the quality of the electrolyte. For this reason, chemically pure substances should be used.

2 Although the electrolytes used are mostly simple salts, tests of the stability of their solutions, which need to be carried out rapidly, often prove not very meaningful. Electrolyte solutions should therefore be discarded at the latest one week after their preparation.

5 TEST PARAMETERS

The parameters chosen for the test, including dissolving, storage and filtration temperature, electrolyte chosen and its concentration, will depend on the type of dyestuff being tested. Examples of combinations of test parameters are given in Table1.

Table 1 Examples of Combinations of Test Parameters

(*Clauses* 4.9 *and* 5)

Dyeing Process	Dissolving Temperature of Dye °C	Filtration/ Storage Temperature °C	Storage Time, *Min*	Electrolyte Added and Concentration of Stock Solution
Batchwise	90	40	120	Sodium chloride, 50 g/l
	60	25	30	Sodium chloride, 60 g/l Sodium carbonate ($Na_2CO_3.10H_2O$), 5g/l
	50	50	0	Sodium chloride, 60 g/l
	80	25	60	Sodium chloride, 50 g/l Sodium carbonate, 5 g/l Sodium hydroxide, 2 ml/l prepared from solution containing approximately 435 g/l
	90	40	90	Sodium chloride, 50 g/l Sodium carbonate, 5 g/l Sodium hydroxide, 2 ml/l prepared from solution containing approximately 435 g/l
Continuous	90	25	30	Sodium hydroxide, 50 g/l Sodium hydroxide, 30 g/l prepared from solution containing approximately 435 g/l
	90	25	120	Sodium carbonate ($Na_2CO_3.10H_2O$), 40 g/l
	90	25	60	Sodium silicate, 95 g/l prepared from solution containing between 34 percent and 40 percent sodium silicate Sodium hydroxide, 38 g/l prepared from solution containing approximately 435 g/l
	90	25	30	Urea, 100 g/l Sodium carbonate ($Na_2CO_3.10H_2O$), 30 g/l

6 PREPARATION OF SOLUTIONS

6.1 The concentrations at which the test dye solutions are prepared shall be chosen considering the expected solution stability limit of the dye in the presence of electrolyte :

Expected Limit to Fall Between	*Stepwise Increase in Dye Concentration Approaching Limit*
1 g/l to 10 g/l	1 g/l
10 g/l to 30 g/l	5 g/l
30 g/l to 100 g/l	10 g/l

The maximum dye concentrations used for the test shall be:

30 g/l for batchwise dyeing processes;and

100 g/l for continuous dyeing processes.

6.2 Place a known amount of dye to be tested in wide-mouthed Erlenmeyer flask and add 150 ml of distilled or fully demineralised water at about 60°C. (but not above the dissolving temperature). Swirl the flask by hand until the dye is fully wetted out (predissolving). Place the solution into the heating bath set at desired dissolving temperature and stir for ten minutes at 500 rev/min to 600 rev/min. In order to stimulate dyebath compositions used in commercial practice , additions of other components to the test solutions are permissible (*see* dyestuffs manufacturer's recommendations). However, these additions, including their influence on volume of test solution shall be noted in the test report. On the other hand changes in volume attributable to changes in temperature should not be taken into account.

Urea as dissolving agent may be added in accordance with the recommendations of dye manufacturers. According to general experience, amount of water should be reduced by 1 ml per gram of urea added, so as not to change the total volume of solution.

6.3 Adjust the dye solution to the desired temperature for filtration or storage using the thermostatically controlled water bath. Add 50 ml of the electrolyte solution (Stock solution) previously adjusted to the test temperature and homogenize the resulting solution.

7 STORAGE

Allow the final test solution to stand at constant temperature for the specified storage time (*see* Table 1).

8 FILTERING THE SOLUTIONS

8.1 Pre-heat the Nutsch filter to the test temperature by pumping through the heating medium from the thermostatic device, and maintain at this temperature throughout the entire filtration operation.

8.2 Immediately before filtering, wet out two filter papers in the Nutsch filter in a double layer with at least 50 ml water at the test temperature.

8.3 Adjust the vacuum to between 3 kPa to 4 kPa (equivalent to 300 mm to 400 mm water column pressure) before filtration so that 200 ml water at the test temperature passes through the filters in a flow-through time of 5-8 seconds.

8.4 Before filtering, homogenize the test solution by swirling it in the flask (visual check). Filter the entire test solution (do not decant) at the desired temperature. Measure the flow-through time with a stopwatch.

8.5 If the solution does not filter within 2 min at a stabilized vacuum, filter it for an additional 2 min under full vacuum.

8.6 After the solution has passed through, continue to extract the filters uniformly under full vacuum for 1 min.

8.7 Allow the filters to dry completely at room temperature before evaluation.

9 EVALUATION

9.1 Compare visually the dried filter paper after filtration of the various test solutions of known concentration. The solubility limit or solution stability limit in the presence of electrolyte is taken as that concentration at which filter residues are seen. Residues that are difficult to see may possibly be detected by gently rubbing the filter surface with a finger tip.

9.2 Flow-through time may be used as a further evaluation criterion. A sudden sharp increase in the flow-through time when moving up the range of solution concentrations indicates that the solubility limit has been exceeded or that the solution is no longer stable.

10 REPORT

10.1 The test report shall include the following:

a) Full identification of the dyestuff under test;

b) Full identification and amount of added electrolyte(s);

c) Type of filter paper used and its manufacturer;

d) Solution stability in the presence of electrolyte, expressed in grams per litre, including the dissolving and storage temperatures;

e) Flow-through time, where applicable;

f) Any special observations during the test or evaluation procedure; and

g) Any deviation, by agreement or otherwise, from the procedure specified (for example total volume of solution other than 200 ml, addition of urea or other components, etc).

ANNEX A

(*Foreword*)

FACTORS AFFECTING RESULTS

The test method has been successfully used in monitoring the quality of dyes for sometime. However, since there are many test variants that depend on production and application methods used, the electrolyte stability data given in the literature is not easy to evaluate. Also, with electrolyte stability different test conditions may lead to different results.

In addition to the factors already known to influence solubility testing without electrolyte, such as filter quality, dissolving temperature, storage temperature and storage time, here the influence of electrolyte must be taken into account. This includes such factors as electrolyte purity and ageing of electrolyte stock solution. In order to increase comparability and reproducibility, the test should be carried out with chemically pure substances, which are normally not used in works practice. Depending on the type and amount of impurity, the values found in evaluation with salts of technical quality may be widely scattered.

Ageing of the stock solution will of course affect laboratory tests only, since in actual practice such solutions are hardly used, certainly not the solutions that have been stored for any length of time. However, in the laboratory the highly concentrated solutions that are mostly used may, with prolonged storage. crystallize out or become chemically changed.

For the sake of simplifying the work, fresh solutions as a rule are not used for each test; however, the age of the solutions is limited, for the reasons given above In cases of dispute, fresh solutions should always be prepared.

It should be borne in mind that the occurance of unexpected interaction between the dye and the electrolyte is possible, and this may lead to equivocal results. Thus, for example, with a particular high concentration of electrolyte and a low mass of dye, precipitation may occur that does not take place with high masses of dye.

Although many process variants are possible in dyeing with reactive dyes and production dyeing conditions are often not comparable with laboratory conditions. this test provides useful indications about the sensitivity of individual reactive dyes to the presence of electrolytes.

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